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Laser Synthesis of Iron Oxide Nanowires

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Iron oxide nanowires are rapidly synthesized in large quantities at room temperature by pulsed-laser (248 nm) ablation of iron powder under methanol. The morphology depends on the rate of collection. At high collection rates, a lamellate 'nanobelt' morphology is observed, whereas at low collection rates nanowires dominate. The as-synthesized products have the stoichiometry of the goethite [FeO(OH)] phase which after annealing at temperatures above 400 °C crystallizes into hematite (α-Fe_2O_3).

Key Words: Nanomaterials, iron oxide nanowires

INTRODUCTION

Nanosized materials exhibit different chemical and physical properties compared to that of bulk form.[1] Iron oxides are widely used in the fields of semiconductor, record material, light catalyst etc.[2] Goethite [α-FeO(OH)] is a common iron oxide and ingredient of iron rust. Hematite (α-Fe_2O_3) is one of the most accepted phases for research due to its novel chemical and physical properties. Conversion of goethite to hematite has many technological applications.[3]

Hematite nanowires are synthesized by different techniques like surface oxidation, thermal oxidation, electrochemical deposition etc.[4] Most of these are complicated processes taking a long time (1.5-120 h), needing different gas flow and high temperature (400-800 °C). Therefore, there is a necessity for fabrication of nanowires of these important oxides in the least complicated procedure with minimum time. In this direction, we have prepared the goethite/hematite nanowires with a very simple technique by laser ablation with the shortest time (5 min). The present article reports the laser synthesis of iron oxide nanowires.

EXPERIMENTAL

Iron oxide nanowires were synthesized by ultraviolet (UV) photochemical technique using the laser pulses from a Lambda-Physik LPX 210i excimer laser operating at 248 nm with a pulse duration of 25 ns.[5] Iron (Fe) powder of maximum particle size 60 μm was used as the starting material. It was placed in an open glass vessel mounted on an X-Y translation motorized stage. Sufficient powder was taken to make a thin layer (~ 5 mm) on the bottom of the vessel. Then it was filled with different liquids (water, ethanol, methanol, isoproponal

and glycol) up to a height of ~ 2 cm. 120 mJ laser pulses were focused to produce a rectangular spot and fluence of 11 J/cm^2. The laser spot was raster scanned over the whole bottom area of the vessel once every 15 s. A laser shot repetition rate of 20 Hz was used throughout the experiments. Iron oxide nanowires were obtained only with methanol. However, the other liquids resulted in solution of nanoparticles. The following may be the possible reactions of goethite nanowire formation.

$$2Fe+12CH_3OH \rightarrow 6C_2H_5OH+2Fe(OH)_3 +3H_2$$

and finally $$Fe(OH)_3 \rightarrow FeO(OH) + H_2O.$$

The starting methanol was clear and colourless. At the beginning, the iron oxide nanobelts were formed when the laser of 248 nm was scanned for 5 minutes and the colour became cloudy yellowish. If it was collected and annealed with this laser for another 10 minutes, it became clear yellow. It should be mentioned that if the rusting was continued to 15 minutes instead of 5 minutes, we got the same yellow colour solution. The yellow solution was dried on glass substrate as well as silicon wafer and annealed at a temperature of 100-800 ^oC. The structure of the iron oxide nanowires/nanobelts and nanoparticles was investigated by FEI Quanta 200 environmental scanning electron microscope (ESEM) and Philips CM200 high-resolution transmission electron microscope (HRTEM).

RESULTS AND DISCUSSION

Figure 1 shows SEM image of iron oxide nanoribbons when rusted in methanol for 5 minutes. If the rusting was made for more than 10 min, we got all circular shaped nanowires as displayed in Fig. 2. A possible mechanism of the formation of nanowires from nanoribbons is as follows. Initially, the nanoribbons are formed with smooth edges when laser shot is run for 5 min.

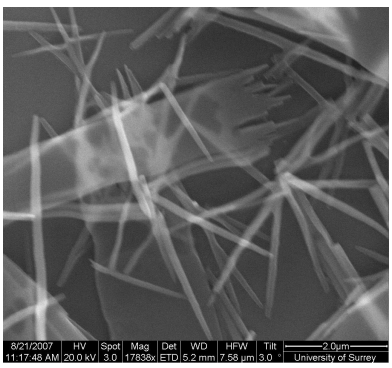

Fig. 1. SEM image of nanoribbons when rusted for 5 minutes.

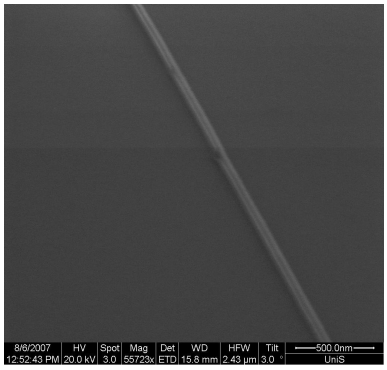

Fig. 2. SEM image of nanowires When rusting is ~ 20 min.

When the laser annealing is increased to ~ 10 min, the edges of the ribbons start fragmented. The wider ribbons are disintegrated into much smaller width ribbons. Ultimately, these turn into round shaped nanowires when laser annealed for ~ 20 min (Fig. 2). It should be mentioned that the iron oxide nanowires were found to be stable in room temperature and air for several months which was very important for their application point of view. The ~ 20 min laser annealed solution is coated on glass slides/silicon substrates and annealed at different temperatures of 100-800 ^{o}C for 2-4 h in air using a tube furnace. The as-produced wires demonstrated circular shape and amorphous nature.[5] When it was annealed at 400 ^{o}C, it started crystallizing. Nevertheless, annealing at 800 ^{o}C crystallized the nanowires fully.[5] Selected area electron diffraction (SAED) pattern of as-produced iron oxide nanowires, showed its amorphous nature with broad and diffuse rings.[5] However, the SAED pattern of 600 ^{o}C annealed samples clearly exhibited the crystallization with sharp and thin rings. Therefore, from TEM and SAED images, it may be concluded that the annealing in air above 400 ^{o}C, crystallized the samples. We found that the as produced nanowires were α-FeO(OH) and amorphous in nature. But the annealing made them crystalline and converted them into α-Fe_2O_3 with the reaction 2 α-FeO(OH) = α-Fe_2O_3 + $3H_2O$.

Conclusions

UV pulsed laser irradiation (248 nm) of iron powder in methanol produced solution of α-FeO(OH) nanowires. Nanoparticles were obtained in other media like water, ethanol, isopropanol, and glycol. Crystalline α-Fe_2O_3 nanowires were obtained by annealing of α-FeO(OH) nanowires.

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